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The Solar Propulsion Group (SPG) at Phillips Laboratory will determine the efficiency of transferring heat from sunlight to a gas using carbon wafers. There will be a series of tests using a calorimeter containing porous carbon and hafnium carbide coated carbon wafers that will absorb the sunlight and transfer the heat to the gas passing through them. We had half the wafers coated with hafnium carbide to prevent carbon loss in a hydrogen rich atmosphere at high temperatures. They will degrade with each test if they are not suitably coated. We conducted a wafer bake-out test to determine if they were suitable for the calorimeter testing, and to develop a baseline for degradation checks to be conducted throughout the calorimeter test series. The preliminary results indicate that the wafers will be unaffected by the environment in the calorimeter. We developed a weight baseline to use in checking the integrity of the wafers throughout the calorimeter testing. The experiments talked about in this paper are not bi-modal in the strictest sense; a single unit supplying both power and propulsion to the spacecraft or satellite. However, in the future, we may find the3 solar/laser thermal propulsion system that the wafers fit into lends itself to both power and propulsion in a single unit. Therefore, the information is pertinent to this forum.

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EVALUATION OF HAFNIUM-CARBIDE WAFERS FOR USE IN A SOLAR CALORIMETER

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Abstract

The Solar Propulsion Group (SPG) at Phillips Laboratory will determine the efficiency of transferring heat from sunlight to a gas using carbon wafers. There will be a series of tests using a calorimeter containing porous carbon and hafnium carbide coated carbon wafers that will absorb the sunlight and transfer the heat to the gas passing through them. We had half the wafers coated with hafnium carbide to prevent carbon loss in a hydrogen rich atmosphere at high temperatures. They will degrade with each test if they are not suitably coated. We conducted a wafer bake-out test to determine if they were suitable for the calorimeter testing, and to develop a baseline for degradation checks to be conducted throughout the calorimeter test series. The preliminary results indicate that the wafers will be unaffected by the environment in the calorimeter. We developed a weight baseline to use in checking the integrity of the wafers throughout the calorimeter testing. The experiments talked about in this paper are not bi-modal in the strictest sense; a single unit supplying both power and propulsion to the spacecraft or satellite. However, in the future, we may find the solar/laser thermal propulsion system that the wafers fit into lends itself to both power and propulsion in a single unit. Therefore, the information is pertinent to this forum.

INTRODUCTION

The Solar Propulsion Group (SPG) at Phillips Laboratory will determine the efficiency of transferring heat from sunlight to a gas using carbon wafers. The wafers in question are supposed to be chemical vapor-deposited (CVD) hafnium carbide on carbon blanks, 190 mm in diameter, uniformly coated to a density of 8% throughout. There is a total volume of 2.019E3 mm^3, where $Volume = L*\pi*d^2/4$ mm^3 of wafer material divided into 12.7 mm. 25.4 mm, and 50.8 mm lengths. The overall length of material was 457.2 mm.

We want to determine the integrity of the wafers *before* subjecting them to a hot hydrogen gas environment. This is impossible. The next best thing is to determine with certainty whether the wafers degrade in the hot hydrogen environment *after* the experiments, then figure out exactly why.

There will be a series of tests using a calorimeter containing porous carbon wafers that will absorb the sunlight and transfer the heat to the gas passing through them. Concentrated and focused sunlight from a primary reflector is directed through an aperture on the front end of what is a water-filled calorimeter or black body cavity. It is in most respects a thruster, except there is no propulsive nozzle at the aft end.

The sunlight must also pass through a window on the front of the calorimeter. Inside the cavity is a stack of porous wafers. The pores have rounded walls. The radiant energy is supposed to hit the rounded pore walls, bounce off at oblique angles toward other pore walls, and after a time, find their way to the aft end of the cavity. This is called radiation trapping. The hydrogen gas, our propellant, is then heated and exhausted out the aft end throught the ejector port.

The wafers are the heat exchange medium. See Fig. 1 below. They are supposed to get very hot; up to 4073 K or greater.

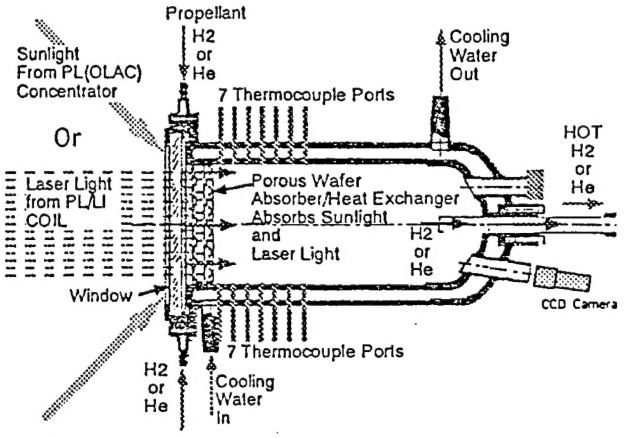

FIGURE 1. Reticulated Vitreous Carbon Calorimeter

The hafnium carbide is supposed to prevent carbon loss in a hydrogen rich atmosphere at high temperatures. The wafers will degrade with each test if they are not suitably coated. The group conducted a bake-out test on the wafers to determine if they were suitable for the calorimeter testing; and to develop a baseline for degradation checks to be conducted throughout the calorimeter test series. We developed a weight baseline to use in checking the integrity of the wafers throughout the calorimeter testing. The preliminary results indicate that the wafers will be unaffected by the environment in the calorimeter.

*

It is important that the wafers do not change composition throughout the test. If the material is not chemically stable in a hot hydrogen environment it is possible that the calorimeter testing could remove material from the wafers. It is also important that the pores of the wafers are not deformed by the heat during the calorimeter tests. Any change in the pores will affect the flow of gases through the wafers (Tuffias 1993). If either of these phenomena occurs, the response of the wafers will vary from one test to the next.

The hot hydrogen may reduce the carbon in the wafers to methane gas. The way we will spot this degradation is to monitor the weight of the wafers before and after each test. This monitoring requires a baseline consisting of the weight of each wafer measured before any of the testing begins. In order for the baseline to be accurate, any contaminants that might affect the weight of the wafers must be removed before the wafers are weighed (Pacquette 1990).

We reduced the magnitude of the wafer weighing by first baking the wafers at medium high temperature in a vacuum. This caused the wafers to outgas. If the wafers are stable just before testing, the only outgassing will be from contaminants and moisture absorbed by the wafers during the transition from the baggie to the calorimeter. Hopefully, exposure will be limitied to a matter of seconds. When we remove the volatile contaminants, the outgassing will stop. The wafers will then be ready to be weighed to establish the baseline. We evaluated the wafer surface optically before and after the heating to see if the pores had been affected.

This paper will describe the test facility, procedures followed, and preliminary data obtained from weighing, evacuating, baking, and then weighing again the hafnium carbide coated carbon wafers between every calorimeter test.

OBJECTIVE

There is one very important goal. It is to determine with certainty whether the wafers we have are completely coated with hafnium carbide throughout its matrix. This means every ligament external and internal has to be coated evenly. It also means there can be no cracks that occurred after the CVD process. The reason is that if hot hydrogen gas impinges on any uncoated part of any wafer during calorimeter testing, the wafer will degrade. This will

cause a change in the weight of the wafer and change the shape of the pores. This will cause inconsistencies in the test data.

BACKGROUND

We think the wafers may not be completely coated with hafnium carbide for the following reasons. a) We visited several vendors before purchasing any wafers. Even the procurement vendor had shown us smaller scale examples that appeared consistent in color, texture, and optical properties with the other vendors. b) A contractor working with us under a Cooperative Research and Development Agreement (CRADA) had difficulties with their procurement that was similar to ours and with the same vendor.

c) Our finished wafers did not resemble samples obtained from another vendor under a different project (DelaRosa and Tuffias 1993). The wafers are supposed to look evenly colored--a taupe (gray-brown) color consistently throughout. All ligaments are supposed to look coated all the way through. The light refraction was supposed to be uniform looking across an exposed planar surface. They are supposed to be devoid of cracks (sources of hydrogen permeation and degradation). The delivered wafers as a whole had none of the above characteristics. They were mottled in appearance. The coloring ranged from very light ashy gray to brown to dark charcoal gray. A showerhead pattern appeared on some, indicating the optical properties were not consistent. And several of the wafers had cracks. We received at least one that was broken in half. The above defects were our first clues that we might not have gotten what we asked for. See the following figures.

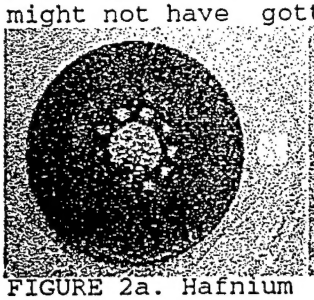

FIGURE 2a. Hafnium Carbide Wafer M15 Front

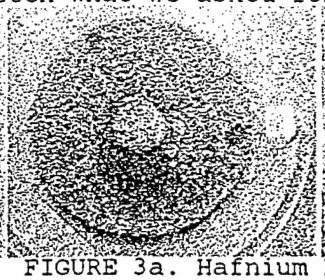

FIGURE 3a. Hafnium Carbide Wafer M13 Front

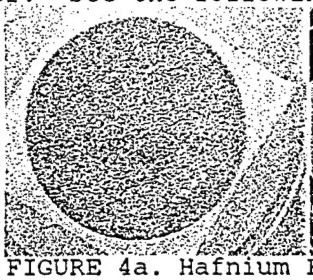

FIGURE 4a. Hafnium Carbide Wafer M11 Front

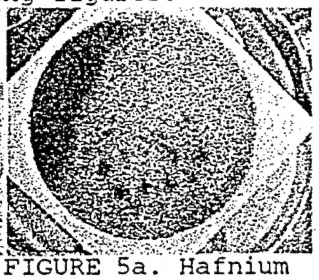

FIGURE 5a. Hafnium Carbide Wafer M1 Front

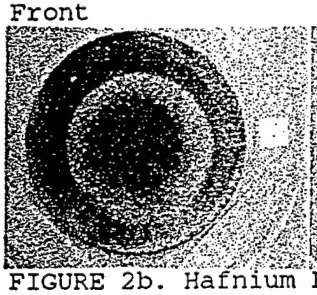

FIGURE 2b. Hafnium Carbide Wafer M15 Reverse

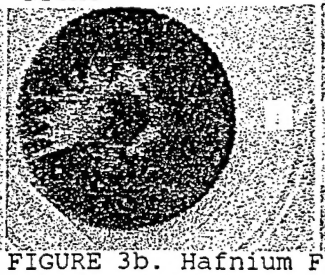

FIGURE 3b. Hafnium Carbide Wafer M13 Reverse

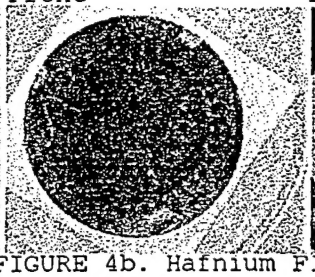

FIGURE 4b. Hafnium Carbide Wafer M11 Reverse

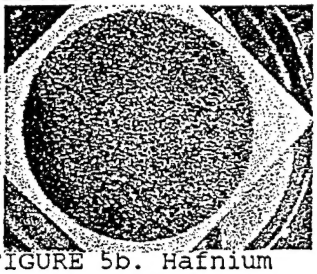

FIGURE 5b. Hafnium Carbide Wafer M1 Reverse

d) We got an expert opinion from Mr. Kamal Updaya at the Phillips Laboratory (PL) who now makes the hafnium carbide coated materials for PL. He stated that the wafers may or may not be coated with hafnium carbide at all, but in any case, are not coated uniformly. Optically, the wafers will present problems with our calorimeter experimental setup. e) We wanted a non-destructive test to determine the wafer material components. There are several special scanning electron microscopes available, but all were being repaired. Also, there were no expert operators that could tell us what we wanted to know if they were tested anyway.

f) We had a thermogravimetric analysis performed on the wafers (Castillo, Capt D.G and Jones, P.F. 1993). We used a wafer with known integrity problems (cracked) for the samples. It was hafnium carbide coated sample number M-7.

The tests showed that percent weight increase is based on the weight of the samples. Percent increase is higher for light samples and lower for heavier samples. We suspect the weight increase is caused by oxygen in the material reacting with nitrogen, or that nitrogen is reacting with impurities in the material. Hafnium Carbide wafer number M7 increased 101.08%.

g) We engaged Wright-Patterson Air Force Base's (WPAFB) Materials Laboratoy's help to determine the content and infiltration of the coating. The results were inconclusive. They used their Computed Tomography (CT) equipment. It produced great clear pictures showing non-uniformity, but could not determine if completely uncoated areas existed or the coating material itself. Hafnium Carbide was not contained in its memory.

h) Dr Mike Holmes of the SPG performed porosity measurements on the wafers. The pore sizes did not match those purported by the vendor for each wafer. Therefore, it is possible other differences between what we asked for, and what we received occurred.

The wafers were supposedly fabricated using the following process or one similar. Porous foams are fabricated by infiltrating a carbon preform that is of the desred shape and size of the final product you want. The CVD reaction, $HfCl4+CH4 \rightarrow HfC+4HCl$, takes place within the carbon preform matrix using standard gas transport techniques involving pressure and/or thermal gradients (DelaRosa and Tuffias 1993).

EXPERIMENTS

Description of the Calorimeter Experimental Hardware

The calorimeter experiments are the reason for going to all the trouble of determining the composition of the wafers and performing the weighing experiments. The calorimeter is made from a 203.2 mm diameter aluminum pipe nested within a 254 mm diameter aluminum pipe. At the aft end, two aluminum end caps cover the pipe ends. At the front end, an annular pipe cap covers the ends. Between the two pipes are aluminum standoffs that keep the two pipes separated from one end to the other and propagate turbulence. This is where the cooling calorimeter water flows. It comes in a pipe through the bottom, flows up and around the 203.2 mm pipe and out the top through another pipe. Fourteen thermocouple ports are drilled through the bottom and top sides, then potted to seal. A hydrogen gas inlet was put in through the top near the front aperture window. Then two pressure transducer ports were put in; one at the front and one near the end. Three pipes were placed through the end; one for the ejector for evacuating the hot hydrogen gas, and two for various instrumentation measurements or cameras. Then the entire calorimeter was jacketed with insulation and wrapped within a thin aluminum split shell. See Fig. 6 and Fig. 7 below. Compare them with Fig. 1.

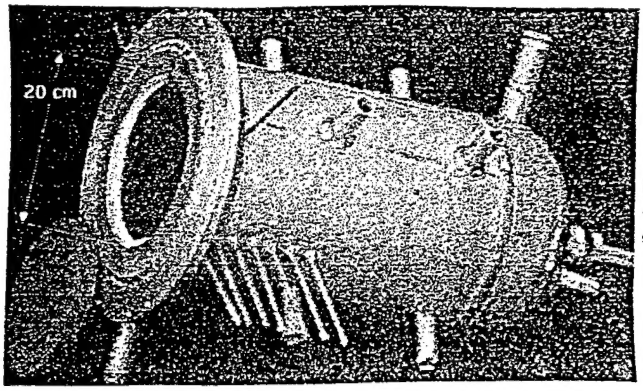

FIGURE 6.Unjacketed RVCC Experimental Hardware Showing Thermocouple Ports

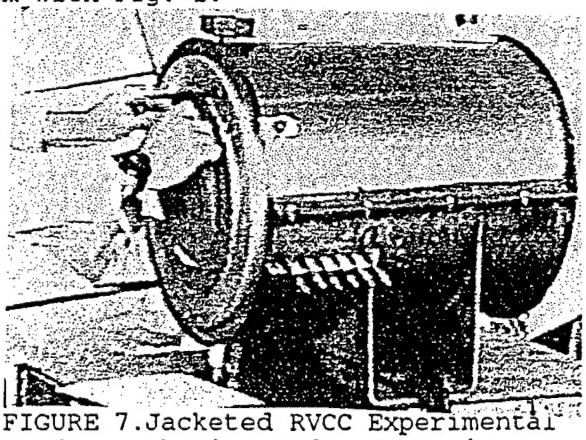

FIGURE 7.Jacketed RVCC Experimental Hardware Showing Wafer Insertion

Description of the Weighing Experimental Hardware

The test hardware consists of the following items. A large bell jar attached to a rough vacuum pump for evacuating several wafers at a time down to 1 millitorr. That was used to speed up the baking process by performing a "cold outgassing" of wafers before baking them in the vacuum oven. After the rough vacuum they were further evacuated singly and then heated to 548.15 K. Then we used the helium purge system to bathe the wafers in a neutral gas at 133.32E13 N/m^2. The initial plan called for an oven with a temperature of 800.15 K but there was no vacuum oven available that met this requirement (Pacquette 1990).

PROCEDURES

Extreme care had to be used when handling the wafers because the surfaces are unusually sensitive to handling. Because of the brittleness of the material, small ligaments break off easily. We took extra care to ensure we do not deposit contaminants on the wafers. Any deposits left on the wafer can skew the test results and cause possible damage to the wafers by acting as heat absorbers. These heat absorbers could create localized hot spots that might erode the wafer surface. Because of this, we wore surgical gloves and face masks whenever working around the wafers. The following initial preparation procedure was followed for each wafer:

Remove the wafer from its protective plastic bag. Measure the wafer's diameter and thickness at three different points along the circumference using a digital caliper accurate to one thousandth of an inch. Record the measurements. Weave a short five millimeter diameter index wire into the top edge of the wafer. The wafers have to be indexed so that they can be correctly oriented when mounted in the calorimeter. The wires are the only practical way to do this. Other methods will either damage and/or contaminate the surface of the wafer.

After the initial preparation we weighed each wafer three separate times to an accuracy of one thousandth of a gram and then repackaged the wafer in its plastic bag. Because the vacuum oven can only hold two wafers at a time, the initial plan was to bake two wafers at a time until outgassing stopped. After the first pair, we found this approach to be too time consuming. We learned that it would take approximately four days to complete the procedure for each pair. With 37 wafers it would take 76 days to bake out all the wafers. We discovered that if a large group of wafers was pulled down to a 133.32×10^{-3} N/m^2 vacuum in a large bell jar before putting pairs in the oven, the total time was reduced to 30 days.

The bell jar procedure is as follows: Carefully remove six wafers from their protective bags. Place the six wafers in the bell jar. Use the roughing pump to pull a vacuum of 133.32×10^{-3} N/m^2. When the vacuum is reached, back fill with gaseous helium. Remove each wafer and carefully place it in its protective plastic bag. This took two days for each group for a total of 12 days. See Fig. 8.

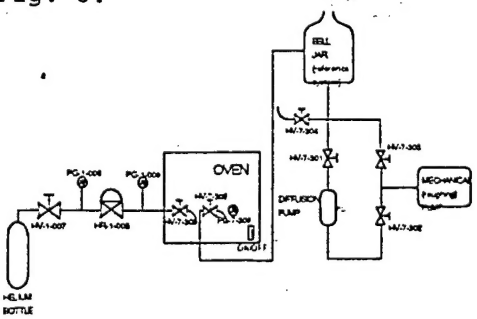

FIGURE 8. Vacuum Setup

The outgassing of the wafers was then completed in the vacuum oven using the following procedure: Carefully remove two wafers from their protective bags and weigh each one three separate times to an accuracy of one thousandth of a gram Place the two wafers in the oven. Using the vacuum system, pull a vacuum of 133.32×10^{-3} N/m^2. Cut off the vacuum. Fill oven with helium until 98.2k N/m^2 vacuum is reached. Turn on the oven, and heat the wafers until they reach 548.15 K. Turn on vacuum system. Pull a vacuum of 133.32×10^{-3} N/m^2.

Turn off the heater and let the oven cool to 303.15 K. Using the helium purge, let the pressure increase to atmosphere. Ensure that the temperature does not decrease faster than 20 K/sec. This was to prevent any damage that might be caused by thermal shock to the wafers before calorimeter testing. Remove the wafers from the oven and weigh them three separate times and record the results. Place each wafer in its plastic bag. It only took one day for each pair to complete the oven bake out following the bell jar procedure for a total of 18 days. Combining the bell jar and vacuum oven procedures resulted in a completion time of 30 days. Another experiment was conducted in which a baked carbon wafer was placed in a plastic bag containing a small vial of water. After several hours the wafer absorbed approximately 1.392 grams of water. As a precaution, we double bagged all the wafers to prevent any absorption of moisture from the air.

RESULTS

See Tables 1 (hafnium carbide) and 2 (carbon). They show the results of the pre and post baking measurements. The post bake measurements are the baseline data that will be used to verify the integrity of the wafers during the calorimeter testing. The diameter, thickness, pre and post bake weight values in the tables are the averages of the measured values. The last column in the tables is the percentage of weight loss. The hafnium carbide wafers have a mean loss of .098699 percent with a standard deviation of .05144. The carbon wafers lost 7.748 percent with a standard deviation of 2.586. The SPG created digitized video images of the wafers before and after the baking to use in a comparison analysis to detect any changes in the wafer surface. The initial results show no substantial change in the wafer pores.

TABLE 1. Hafnium Carbide Wafers

Wafer	Diameter (cm)	Thickness (cm)	Pores per Inch	Bell jar weight (grams)	Prebake weight (grams)	Postbake weight (grams)	Difference (grams)	Percent weight (%)
M-1	18.818	1.270	20	271.036	270.756	270.538	0.498	0.184
M-2	19.020	1.223	10	577.982	577.576	577.356	0.626	0.108
M-3	18.678	1.333	65	302.302	302.182	301.987	0.315	0.104
M-4	18.871	1.268	45	304.905		304.714	0.191	0.063
M-5	18.921	1.232	100	324.528	324.455	324.210	0.318	0.098
M-6	18.882	1.277	80	283.603		283.371	0.232	0.082
M-7	18.920	2.529	20	634.902	634.303	634.035	0.867	0.137
M-8	18.750	2.531	45	571.778	571.182	571.062	0.716	0.125
M-9	18.815	2.535	65	580.815	580.650	580.561	0.254	0.044
M-10	18.794	2.548	80	567.106	567.035	566.939	0.168	0.030
M-11	18.773	2.551	45	676.544	676.063	675.824	0.720	0.107
M-12	18.681	2.552	100	570.919	570.851	570.788	0.131	0.023
M-13	18.765	2.525	20	573.279	572.770	572.060	1.219	0.213
M-14	18.818	2.54	20	583.422	582.705	582.612	0.810	0.139
M-15	18.684	2.278	45	574.245	573.894	573.594	0.650	0.113
M-16	18.800	1.223	100	297.006	296.935	296.923	0.082	0.028
M-17	18.648	2.513	65	648.058	647.496	647.438	0.619	0.096
M-18	18.680	2.544	80	559.497	559.173	559.031	0.466	0.083

M-4 and M-6 prebake data is not available.

TABLE 2. Carbon wafers

Wafer	Diameter (cm)	Thickness (cm)	Pores per Inch	Bell jar weight (grams)	Prebake weight (grams)	Postbake weight (grams)	Difference (grams)	Percent weight (%)
C-1	19.055	5.043	100	82.607	81.291	78.852	3.755	4.762
C-2	19.054	2.548	100	39.464	37.674	36.679	2.785	7.594
C-3	19.047	1.269	100	19.483	19.483	18.339	1.144	6.238
C-4	19.046	2.559	80	40.912	37.480	37.151	3.761	10.125
C-5	19.036	2.548	80	46.854	42.630	42.316	4.539	10.726
C-6	19.015	1.276	80	19.924	18.233	18.052	1.872	10.372
C-7	19.032	2.541	60	35.095	32.190	31.546	3.549	11.251
C-8	19.039	2.541	60	36.056	33.185	32.786	3.270	9.975
C-9	19.048	2.536	60	44.233	41.059	40.320	3.913	9.705
C-10	19.029	1.334	60	22.833	20.994	20.566	2.267	11.023
C-11	19.060	2.541	45	48.749	46.294	45.328	3.421	7.547
C-12	19.045	2.521	45	44.413	42.432	41.271	3.142	7.613
C-13	19.047	2.540	45	44.751	42.801	41.650	3.101	7.445
C-14	19.049	1.271	45	21.487	20.408	19.910	1.577	7.919
C-15	19.045	2.526	20	41.818	41.657	40.143	1.675	4.173
C-16	19.031	2.515	20	36.583	36.472	35.027	1.557	4.444
C-17	19.030	2.535	20	39.674	38.212	37.718	1.956	5.166
C-18	19.020	1.211	20	19.296	19.296	17.804	1.492	8.380
C-19	19.040	2.522	10	37.372	36.325	36.377	0.995	2.734

CONCLUSIONS

The weight loss shows that the wafers did have volatile contaminants that could alter the calorimeter testing results. It is imperative that a wafer bake out be conducted before and after each calorimeter test. We suspect the weight loss for each wafer is a combination of moisture and contaminants from wafer processing. The weight changes are consistent with similar testing performed by the Composites Laboratory at the Phillips Laboratory, Edwards AFB.

The results show that an outgassed wafer will readily absorb material from its environment. Therefore, it is essential that the wafers be properly handled during the bake out, and that they are properly packaged to prevent the absorption of material from the environment.

The re-absorption raises some concerns for the calorimeter testing. Ideally each calorimeter test should begin immediately after the installation of the wafers and the wafers should be weighed as soon as the test is completed. Safety and other practical considerations prevent us from meeting this ideal. This will complicate our monitoring of potential wafer degradation.

All indications are that the structural integrity of the wafers will not be adversely affected by the environment of the calorimeter. It is unknown whether they are fully coated hafnium carbide on carbon wafers. They do, however, appear to be suitable for use in the calorimeter testing.

Acknowledgments

We wish to thank Ms. Lisa Barland, Mr. Robert Douglas, Mr. Leonard Langdon, and former SSgt Mark Horn for performing the baseline wafer weighing experiments.

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